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**Correlating uncompensated antiferromagnetic moments and exchange coupling interactions in interface ion-beam bombarded Co<sub>90</sub>Fe<sub>10</sub>/CoFe-oxide bilayers**

Chin Shueh

*National Chung Hsing University, Taiwan*

Pei-Shi Chen

*National Chung Hsing University, Taiwan*

David Cortie

*University of Wollongong, dlc422@uowmail.edu.au*

Frank Klose

*ANSTO, Frank.Klose@ansto.gov.au*

Wen-Chen Chen

*National Yunlin University Of Science And Technology, Taiwan*

*See next page for additional authors*

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# Correlating uncompensated antiferromagnetic moments and exchange coupling interactions in interface ion-beam bombarded Co<sub>90</sub>Fe<sub>10</sub>/CoFe-oxide bilayers

## Abstract

The coercivity and exchange bias field of ferro-/antiferromagnetic Co<sub>90</sub>Fe<sub>10</sub>/CoFe-oxide bilayers were studied as function of the surface morphology of the bottom CoFe-oxide layer. The CoFe-oxide surface structure was varied systematically by low energy (0-70 V) Argon ion-beam bombardment before subsequent deposition of the Co<sub>90</sub>Fe<sub>10</sub> layer. Transmission electron microscopy results showed that the bilayer consisted of hcp Co<sub>90</sub>Fe<sub>10</sub> and rock-salt CoFe-oxide. At low temperatures, enhanced coercivities and exchange bias fields with increasing ion-beam bombardment energy were observed, which are attributed to defects and uncompensated moments created near the CoFe-oxide surface in increasing amounts with larger ion-beam bombardment energies. Magnetometry results also showed an increasing divergence of the low field temperature dependent magnetization [ $\Delta M(T)$ ] between field-cooling and zero-field-cooling processes, and an increasing blocking temperature with increasing ion-beam bombardment energy. © 2012 The Japan Society of Applied Physics.

## Keywords

correlating, moments, exchange, bilayers, oxide, interactions, coupling, cofe, antiferromagnetic, 10, co<sub>90</sub>fe, bombarded, beam, ion, interface, uncompensated

## Disciplines

Engineering | Science and Technology Studies

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## Authors

Chin Shueh, Pei-Shi Chen, David Cortie, Frank Klose, Wen-Chen Chen, T H. Wu, Johan Van Lierop, and Ko-Wei Lin

Correlating Uncompensated Antiferromagnetic Moments and Exchange Coupling  
Interactions in Interface Ion-Beam Bombarded  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-Oxide Bilayers

Chin Shueh<sup>1</sup>, Pei-Shi Chen<sup>1</sup>, David Cortie<sup>2,3</sup>, Frank Klose<sup>3</sup>, Wen-Chen Chen<sup>4</sup>, Te-Ho Wu<sup>4</sup>, Johan van Lierop<sup>5</sup>, and Ko-Wei Lin<sup>1,\*</sup>

<sup>1</sup>Department of Materials Science and Engineering, National Chung Hsing University,  
Taichung 402, Taiwan

<sup>2</sup>Institute for Superconducting and Electronic Materials, University of Wollongong,  
Wollongong 2522, Australia

<sup>3</sup>Australian Nuclear Science and Technology Organisation, Kirrawee 2232, Australia

<sup>4</sup>Taiwan Spin Research Center, National Yunlin University of Science and Technology,  
Douliu 640, Taiwan

<sup>5</sup>Department of Physics and Astronomy, University of Manitoba, Winnipeg R3T 2N2,  
Canada

\*Corresponding author. E-mail address: kwlin@dragon.nchu.edu.tw

## Abstract

The coercivity and exchange bias field of ferro-/antiferromagnetic  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-oxide bilayers were studied as function of the surface morphology of the bottom CoFe-oxide layer. The CoFe-oxide surface structure was varied systematically by low energy (0 – 70 V) Argon ion-beam bombardment before subsequent deposition of the  $\text{Co}_{90}\text{Fe}_{10}$  layer. Transmission electron microscopy results showed that the bilayer consisted of hcp  $\text{Co}_{90}\text{Fe}_{10}$  and rock-salt CoFe-oxide. At low temperatures, enhanced coercivities and exchange bias fields with increasing ion-beam bombardment energy were observed, which are attributed to defects and uncompensated moments created near the CoFe-oxide surface in increasing amounts with larger ion-beam bombardment energies. Magnetometry results also showed an increasing divergence of the low field temperature dependent magnetization  $[\Delta M(T)]$  between field-cooling and zero-field-cooling processes, and an increasing blocking temperature with increasing ion-beam bombardment energy.

## 1. Introduction

Exchange bias [1-6], i.e., the shift of the hysteresis loop of a ferromagnetic (FM) material in contact with an antiferromagnetic (AFM) material after a field-cooling process, depends on many factors including the particular materials involved [7-10], film growth conditions [11-14], the structural, compositional and magnetic details of the interfaces [15-18], the magnetic stiffness of the AFM moments, and the field-cooling conditions [19-22].

In this work, we varied the structural and magnetic interface between FM and AFM  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-oxide bilayers by bombarding the surface of the bottom AFM CoFe-oxide layer with Argon ions of varying energy before the subsequent growth of the top FM  $\text{Co}_{90}\text{Fe}_{10}$  layer. We observed a systematic increase of the coercivity and exchange bias at low temperature as function of the Ar ion-beam energy, which we attributed to an increase in the creation of defects and uncompensated moments in the AFM layer from higher ion-beam energies.

## 2. Experimental Methods

The  $\text{Co}_{90}\text{Fe}_{10}$  (at%) / CoFe-oxide bilayers were prepared on oxidized Si wafer substrates by using a dual ion-beam sputtering deposition technique [21,22]. A Kaufman ion source (800 V, 7.5 mA) was used to focus an Argon ion-beam onto a commercial  $\text{Co}_{90}\text{Fe}_{10}$  target surface in order to fabricate the top  $\text{Co}_{90}\text{Fe}_{10}$  layer. An End-Hall ion source ( $V_{\text{EH}} = 100$  V, 500 mA) was used to in-situ bombard the growing bottom layer

during deposition with a mixture of 41% O<sub>2</sub> / Ar (O<sub>2</sub>/Ar flow rate: 1.6/2.3 sccm) in order to fabricate the bottom CoFe-oxide layer (17 nm nominal thickness). Before capping with the top Co<sub>90</sub>Fe<sub>10</sub> layer (23 nm nominal thickness), the surface of the CoFe-oxide layer was bombarded by 100% Ar ions using the same End-Hall source. The acceleration voltage (V<sub>EH</sub>) was varied from 40 to 70 V for different films in order to create varying surface microstructure on the CoFe-oxide layer.

### 3. Results and Discussion

The microstructures of the Co<sub>90</sub>Fe<sub>10</sub> (~17 nm) / CoFe-oxide (~23 nm) bilayers were characterized by TEM (Transmission Electron Microscopy), as shown in Fig. 1. The un-bombarded (V<sub>EH</sub> = 0 V) Co<sub>90</sub>Fe<sub>10</sub> / CoFe-oxide bilayers were polycrystalline with grain sizes ranging from 5 to 15 nm. The respective electron diffraction patterns [Fig. 1(a)] indicated that the bilayers consisted of hcp Co<sub>90</sub>Fe<sub>10</sub> (a~ 2.5 Å, c~ 4.1 Å) and rock-salt CoFe-oxide (a~ 4.2 Å), in agreement with our previous works [22,23] on these CoFe-based film systems. The bombardment of the bottom CoFe-oxide layer surface with low-energy Ar ion-beams (V<sub>EH</sub> = 40 to 70 V) neither changed the crystal structure nor altered significantly the grain size distribution close to the CoFe-oxide surface, as revealed by the TEM images and diffraction patterns shown in Figs. 1(c) and 1(d) for Co<sub>90</sub>Fe<sub>10</sub> / CoFe-oxide (V<sub>EH</sub>= 50 V) and Figs. 1(e) and 1(f) for Co<sub>90</sub>Fe<sub>10</sub> / CoFe-oxide (V<sub>EH</sub>= 70 V). As can be seen in the cross-sectional TEM images in Figs. 1(b), 1(d) and 1(f), all Co<sub>90</sub>Fe<sub>10</sub> / CoFe-oxide bilayers exhibited a smooth interface with roughnesses

considerably below 2 nm. It can be concluded from the data presented in Fig. 1 that the low-energy ion-beam bombardment did not lead to significant microstructural and morphological variations (lattice constant, grain size, roughness, and interface flatness) in the AF CoFe-oxide layer.

The room temperature hysteresis loops of the different  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-oxide bilayers made with Ar ion-beam bombardment voltages,  $V_{\text{EH}} = 0$  to 70 V, are shown in Fig. 2. Since  $\text{Co}_{90}\text{Fe}_{10}$  is a ferromagnet ( $T_{\text{c, bulk}} \sim 1800$  K [24]) and CoFe-oxide is an antiferromagnet with a Néel temperature less than that of CoO ( $T_{\text{N, bulk}} \sim 290$  K [25]), no exchange bias effects were expected at room temperature. The  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-oxide bilayer without ion-beam bombardment ( $V_{\text{EH}} = 0$  V) exhibited a square hysteresis loop with a high remanence magnetization,  $M_{\text{r}}$ , and a coercivity of  $H_{\text{c}} \sim 15$  Oe, as is shown in Fig. 2(a). Increasing the ion-beam bombardment energy [Fig. 2(b)] resulted in a systematic decrease of  $M_{\text{r}}$ , while  $H_{\text{c}}$  remained unaffected ( $\sim 20$  Oe).

The decrease in  $M_{\text{r}}$  with increasing  $V_{\text{EH}}$  indicates that the ion-beam bombardment of the bottom CoFe-oxide layer may have created defects that influenced strongly the magnetization reversal processes in the  $\text{Co}_{90}\text{Fe}_{10}$  layer. Since the CoFe-oxide layer is believed to be paramagnetic at 298 K, structural defects [26] must have been responsible for the low  $M_{\text{r}}$  values of the ion-bombarded samples.

To study exchange bias effects, the  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-oxide bilayers were 12 kOe field-cooled from 350 to 50 K. The hysteresis loops for the films after field cooling are shown in Fig. 2(c). In contrast to the room temperature behavior, for all  $V_{\text{EH}}$

bombardment energies, a square and symmetric loop shape was present, and a high remanence concomitant with the square loops, i.e.  $M_r / M_s \sim 1$ . Figure 2(c) identifies clearly that an increasing exchange bias field ( $H_{ex}$ ) developed with increasing ion-beam bombardment energy. The dependence of  $H_c$  and  $H_{ex}$  on  $V_{EH}$  [Fig. 2(d)] shows that without ion-beam bombardment,  $H_{ex} \sim -150$  Oe and  $H_c \sim 250$  Oe was measured, and for increasing  $V_{EH}$  values up to 70 V a monotonic increase of  $H_c$  and  $H_{ex}$  resulted. These trends contrast those observed in a previous study on films of similar composition with modifications of the interface [23]. Using much higher ion-beam bombardment energies of  $V_{EH} = 70 - 150$  V, caused greater damage to the AF spin configuration in the CoFe-oxide layer and increased in the degree of misalignment [27,28], which resulted in a decrease of  $H_{ex}$ . In addition, the linear increase of  $H_c$  vs  $V_{EH}$  in the  $Co_{90}Fe_{10} / CoFe$ -oxide bilayers in this study indicates that an enhancement of the  $Co_{90}Fe_{10}$  anisotropy could be achieved by coupling to the defects created by ion-beam bombardment on the AF CoFe-oxide surface that act as pinning sites of domains during the magnetization reversal processes [21].

Further evidence of the nature of the exchange coupling between the  $Co_{90}Fe_{10}$  and CoFe-oxide layer is evident in the temperature dependence of the zero field-cooled (ZFC) and field-cooled (FC) DC susceptibility ( $M$  vs  $T$ ) data measured with a Quantum Design VSM using 100 Oe, as shown in Fig. 3. The difference in the ZFC/FC curves is small (e.g., consider the  $\Delta M_{FC-ZFC}$  in Fig. 4) in  $Co_{90}Fe_{10} / CoFe$ -oxide bilayers without ( $V_{EH} = 0$  V) or with low energy ion-beam bombardment ( $V_{EH} = 40$  and 50 V). However, a



significant increase in the ZFC moment with increasing temperature from 50 to 300 K was observed for the largest  $V_{EH} = 70$  V [Fig. 3]. This parallels the change in the ferromagnetic response as a function of temperature, and is a signature of the exchange coupling between the  $Co_{90}Fe_{10}$  and the CoFe-oxide layer, identified by its larger  $H_{ex}$ . Note that a similar effect was also observed in NiFe/NiFeO thin films [29]. The blocking temperature,  $T_B$ , estimated by the magnetization with a maximum in the ZFC scan [29], was found to increase with increasing  $V_{EH}$  from 180 K ( $V_{EH} = 0$  V) to 230 K ( $V_{EH} = 70$  V), as shown in the inset of Fig. 3.

Our previous work on NiFe/NiO bilayers [21] has shown that the pure ferromagnetic (e.g., permalloy) layer usually exhibited identical ZFC and FC curve with  $H_{app} = 100$  Oe (i.e.  $\Delta M_{FC-ZFC}(T) = 0$ ), while the coupling of the FM layer with an AFM layer like NiO will result in a clear divergence between  $M_{ZFC}(T)$  and  $M_{FC}(T)$ . In the present study, a similar behavior was found in which the degree of divergence ( $\Delta M_{FC-ZFC}$ ) seems to depend strongly on the ion-beam modification of exchange bias, as demonstrated by the increase of  $\Delta M_{FC-ZFC}$  (at 50 K) with increasing  $V_{EH}$ , as shown in Fig. 4.

The degree of exchange coupling is qualitatively estimated by the difference in magnetization ( $\Delta$ ) [29] between FC and ZFC at 50 K, which is about 3% in a spin (or cluster) glass [30] and about 0.5% in a pure permalloy layer [29]. The  $Co_{90}Fe_{10}$  / CoFe-oxide ( $V_{EH} = 70$  V) bilayer exhibited the largest difference in magnetic moment ( $\Delta \sim 40\%$ ), compared to those of  $\Delta$  ( $<10\%$ ) in the  $Co_{90}Fe_{10}$  / CoFe-oxide bilayers with CoFe-oxide surface being bombarded by lower energies (40 and 50 V) or without

bombardment ( $\Delta \sim 3\%$ ). The  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-oxide ( $V_{\text{EH}} = 70$  V) bilayer also had the highest exchange bias, and lowest room temperature remanence  $M_r$ . Therefore, the difference ( $\Delta$ ) in magnetic moment at 50 K between ZFC and FC curves is a measure of the onset of exchange coupling between FM  $\text{Co}_{90}\text{Fe}_{10}$  and AFM CoFe-oxide, which altered the ferromagnetic properties. Moreover, it is clear that the temperature where this occurred depended on the ion-beam bombardment energy, implying the formation of differing amounts of stable uncompensated AF CoFe-oxide moments with differing ion doses.

#### 4. Conclusions

Low-energy Ar ion-beam bombardment was used to modify the exchange bias effects of  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-oxide bilayers. At low temperature, the  $\text{Co}_{90}\text{Fe}_{10}$  / CoFe-oxide bilayers exhibited an almost linear increase of coercivity and exchange bias field with increasing Ar ion-beam bombardment energy on the bottom CoFe-oxide surface. This indicated that the ion-beam bombardment of the CoFe-oxide surface created defects that acted as pinning sites to affect the magnetization reversal processes in  $\text{Co}_{90}\text{Fe}_{10}$ , thus resulting in coercivity enhancements. This process also promoted the formation of thermally-stable uncompensated moments near the surface of the bottom CoFe-oxide layer that permitted exchange bias loop shifts.

## Acknowledgments

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## References

- [1] R. L. Stamps: *J. Phys. D: Appl. Phys.* **33** (2000) R247.
- [2] J. Nogues, J. Sort, V. Langlais, V. Skumryev, S. Surinach, J. S. Munoz, and M. D. Baro: *Phys. Rep.* **422** (2005) 65.
- [3] K. O’Grady, L. E. Fernandez-Outon, and G. Vallejo-Fernandez: *J. Magn. Magn. Mater.* **322** (2010) 883.
- [4] A. E. Berkowitz and K. Takano: *J. Magn. Magn. Mater.* **200** (1999) 552.
- [5] M. Kiwi: *J. Magn. Magn. Mater.* **234** (2001) 584.
- [6] N. C. Koon: *Phys. Rev. Lett.* **78** (1997) 4865.
- [7] D. L. Cortie, K.-W. Lin, C. Shueh, H.-F. Hsu, X. L. Wang, M. James, H. Fritzsche, S. Bruck, and F. Klose: *Phys. Rev. B* **86** (2012) 054408.
- [8] T. Saerbeck, F. Klose, D. Lott, G. J. Mankey, Z. Lu, P. R. LeClair, W. Schmidt, A. P. J. Stampfl, S. Danilkin, M. Yethiraj, and A. Schreyer: *Phys. Rev. B* **82** (2010) 134409.
- [9] J. Dho, C. W. Leung, Z. H. Barber, and M. G. Blamire: *Phys. Rev. B* **71** (2005) 180402.
- [10] M. R. Fitzsimmons, B. J. Kirby, S. Roy, Z.-P. Li, I. V. Roshchin, S. K. Sinha, and I. K. Schuller: *Phys. Rev. B* **75** (2007) 214412.

- [11] H. Bea, M. Bibes, F. Ott, B. Dupe, X.-H. Zhu, S. Petit, S. Fusil, C. Deranlot, K. Bouzehouane, and A. Barthelemy: *Phys. Rev. Lett.* **100** (2008) 017204.
- [12] G. Li, C. W. Leung, C. Shueh, H.-F. Hsu, H.-R. Huang, K.-W. Lin, P. T. Lai, and P. W. T. Pong: *Surf. Coatings Technol.* (in press) [DOI:10.1016/j.surfcoat.2012.05.035].
- [13] K.-W. Lin, V. V. Volobuev, J.-Y. Guo, S.-H. Chung, H. Ouyang, and J. van Lierop: *J. Appl. Phys.* **107**, (2010) 09D712.
- [14] K.-W. Lin, M.-R. Wei, and J.-Y. Guo: *J. Nanosci. Nanotechnol.* **9** (2009) 2023.
- [15] K.-W. Lin, J.-Y. Guo, S. Kahwaji, S.-C. Chang, H. Ouyang, J. van Lierop, N. N. Phuoc, and T. Suzuki: *Phys. Status Solidi A* **204** (2007) 3970.
- [16] T. Ambrose and C. L. Chien: *Appl. Phys. Lett.* **65** (1994) 1967.
- [17] R. Morales, Z. P. Li, J. Olamit, K. Liu, J. M. Alameda, I. K. Schuller: *Phys. Rev. Lett.* **102** (2009) 097201.
- [18] R. Magaraggia, M. Kostylev, R. L. Stamps, K.-W. Lin, J.-Y. Guo, K.-J. Yang, R. D. Desautels, and J. van Lierop: *IEEE Trans. Magn.* **47** (2011) 1614.
- [19] J. Nogues, D. Lederman, T. Moran, I. K. Schuller: *Phys. Rev. Lett.* **76** (1996) 3186.
- [20] Y. Ijiri, T. C. Schulthess, J. A. Borchers, P. J. van der Zaag, R. W. Erwin: *Phys. Rev. Lett.* **99** (2007) 147201.
- [21] K.-W. Lin, M. Mirza, C. Shueh, H.-R. Huang, H.-F. Hsu and J. van Lierop: *Appl. Phys. Lett.* **100** (2012) 122409.
- [22] K.-W. Lin and J.-Y. Guo: *J. Appl. Phys.* **104** (2008) 123913.

- [23] C. Shueh, D. L. Cortie, F. Klose, J. van Lierop, and K.-W. Lin: IEEE Trans. Magn. (in press) [DOI: 10.1109/TMAG.2012.2201144].
- [24] T. B. Massalski and H. Okamoto: Binary Alloy Phase Diagrams (ASM International, Materials Park, OH, 1990).
- [25] R. C. O'Handeley: Modern Magnetic Materials, Principle and Applications (Wiley, New York, 2000).
- [26] K.-W. Lin, R. J. Gambino, and L. H. Lewis: J. Appl. Phys. **93** (2003) 6590.
- [27] J. McCord, C. Hamann, R. Shafer, L. Schultz, and R. Mattheis: Phys. Rev. B **78** (2008) 094419.
- [28] K.-W. Lin, T.-J. Lin, J.-Y. Guo, H. Ouyang, D.-H. Wei, and J. van Lierop: J. Appl. Phys. **105** (2009) 07D710.
- [29] K.-W. Lin, R. J. Gambino, and L. H. Lewis: Jpn. J. Appl. Phys. **44** (2005) 3936.
- [30] S. Mukherjee, R. Ranganathan, P. S. Anilkumar, and P. A. Joy: Phys. Rev. B **54** (1996) 9267.

## Figure captions

Fig. 1. The planar-view TEM micrographs of the bottom CoFe-oxide layer with Ar ion-beam bombardment on the surface with different energies ( $V_{EH}$ ) of (a) 0 V, (c) 50 V, and (e) 70 V. The cross-sectional TEM micrographs of the  $Co_{90}Fe_{10}$  / CoFe-oxide bilayer with the bottom layer bombarded with different  $V_{EH}$  are shown in (b) 0 V, (d) 50 V, and (f) 70 V, respectively.

Fig. 2. Hysteresis loops of  $Co_{90}Fe_{10}$  / CoFe-oxide ( $V_{EH}$ = 0, 40, 50, and 70 V) bilayers measured at (a) 298 K and (c) 50 K (after FC in 12 kOe). (b) Remanent magnetization  $M_r$  at 298 K as function of  $V_{EH}$ . (d) Dependence of  $H_c$  and  $H_{cx}$  on  $V_{EH}$  at 50 K after FC.

Fig. 3. The temperature dependence of ZFC and FC magnetization of  $Co_{90}Fe_{10}$  / CoFe-oxide bilayers. The blocking temperature vs  $V_{EH}$  is shown in the inset.

Fig. 4. The temperature dependence of the difference between ZFC and FC magnetization ( $\Delta M_{FC-ZFC}$ ) of  $Co_{90}Fe_{10}$  / CoFe-oxide bilayers. The  $\Delta M_{FC-ZFC}$  at 50 K vs  $V_{EH}$  is shown in the inset.

Fig. 1.

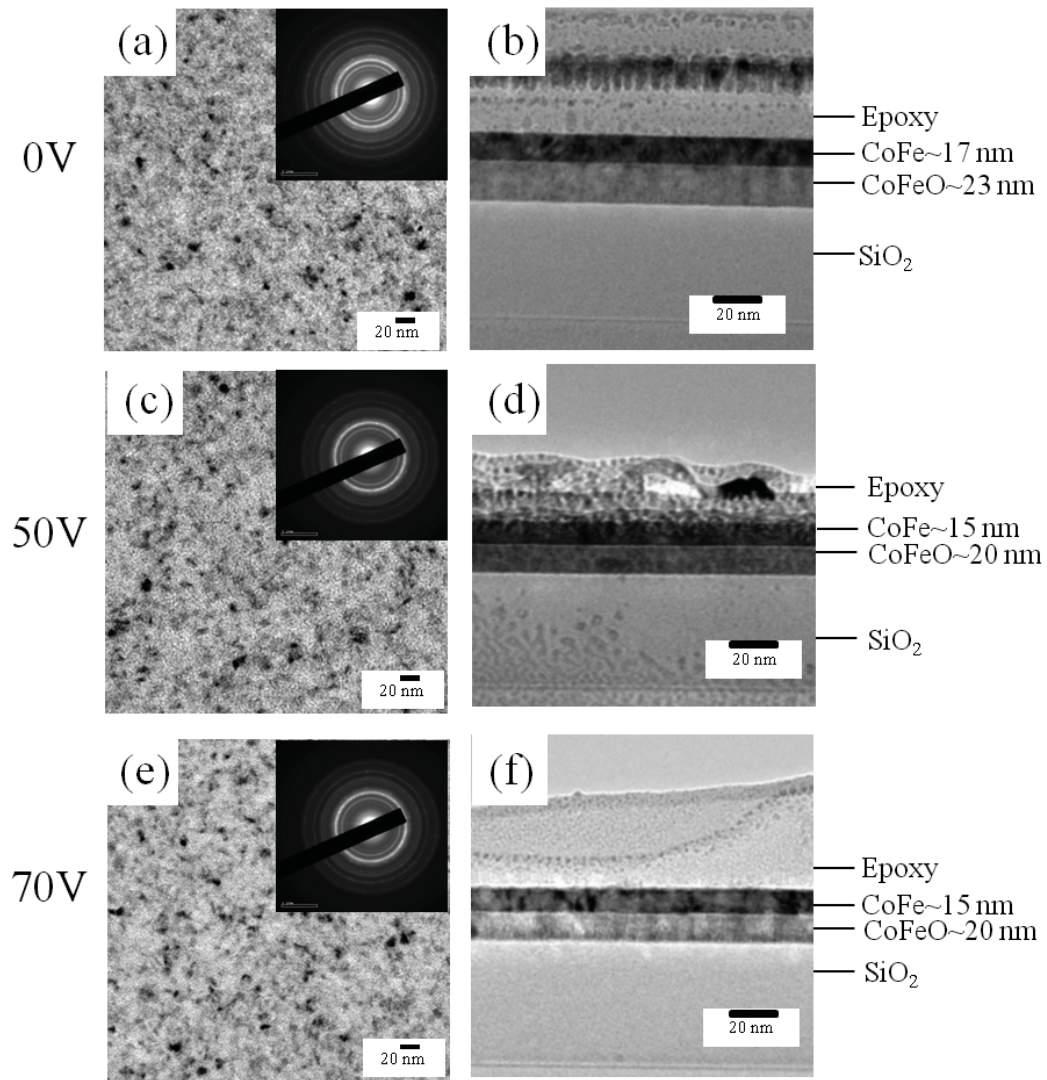


Fig. 2.

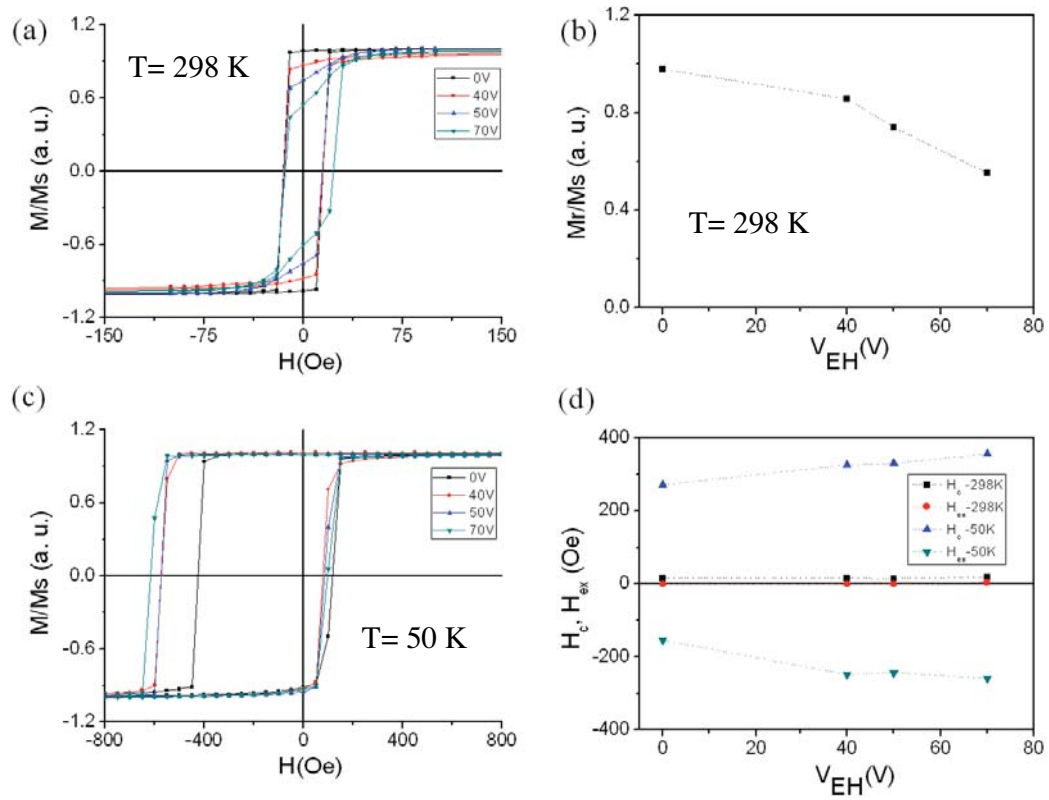




Fig. 3.

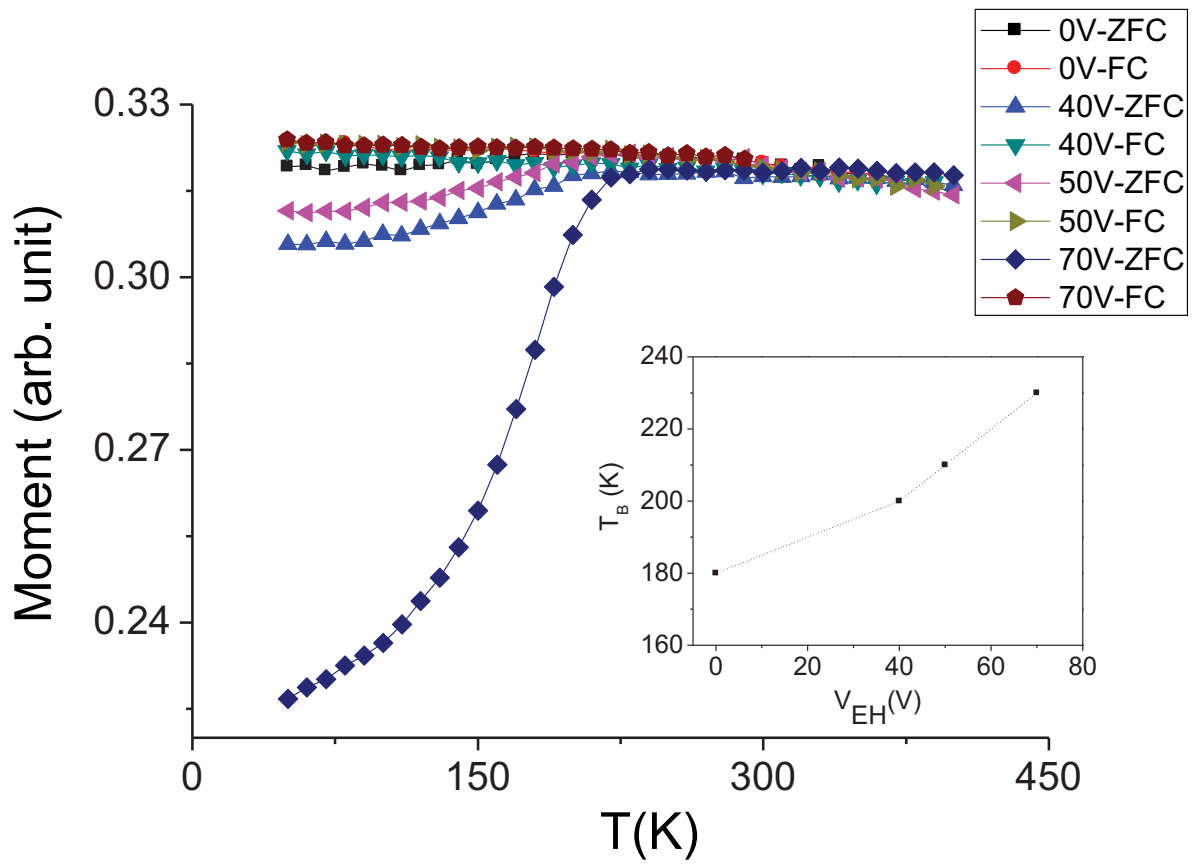


Fig. 4.

